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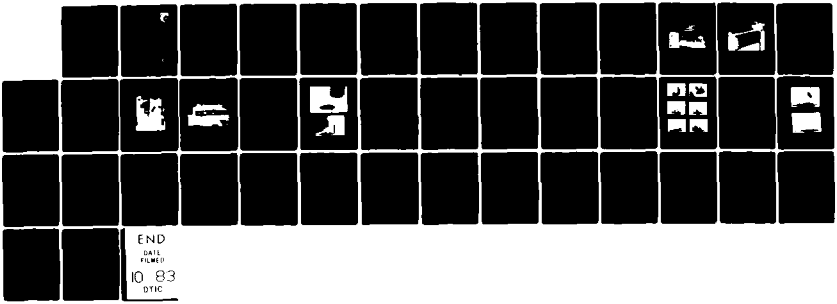

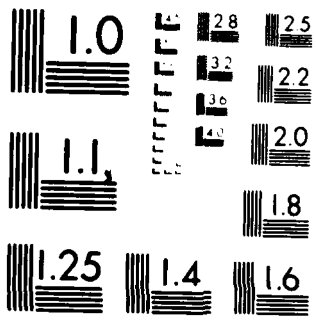

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AFWAL-TR-82-2110

HIGH TEMPERATURE (1649°C, 3000°F) SURFACE IGNITION TEST APPARATUS FOR FLUIDS

Leo Parts and Richard G. Olt

MONSANTO RESEARCH CORPORATION
DAYTON LABORATORY
1515 NICHOLAS ROAD
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MARCH 1983

FINAL REPORT FOR PERIOD 1 NOVEMBER 1980 - 31 MARCH 1982

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This technical report has been reviewed and is approved for publication.

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A high temperature surface ignition test apparatus has been designed, fabricated, and performance-tested. It was designed to determine ignition temperatures and to observe flame propagation characteristics of aircraft fluids at temperatures ranging up to 1649°C (3000°F). A resistance-heated graphite element functions as the hot surface. Its temperature is controlled by electrical power input. The graphite heater simulates graphite composite brake surfaces in high performance aircraft. Results of ignition tests with presently used and developmental fluids are presented.

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FORFWORD

This Technical Report was prepared by Monsanto Research Corporation, Dayton Laboratory. It covers a part of the technical effort under Contract No. F33615-78-C-2023, Project 3048, Task 304807, and Work Unit 30480784, during the period November 1980 - March 1982. Messrs. G. W. Gandee and G. A. Spencer, AFWAL/POSH served as Project Engineers. Dr. Leo Parts of Monsanto Research Corporation was technically responsible for the work.

Messrs. G. W. Gandee and B. P. Botteri, and other members of the Aero Propulsion Laboratory were instrumental in establishing the performance requirements for the apparatus.

Mr. R. G. Olt, coauthor of this report, provided valuable assistance by sharing his years of experience in this project, beyond his retirement date.

Mr. J. D. Arehart assisted materially through fabrication of the apparatus.

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SECTION I

INTRODUCTION

The main objective of the program, within whose scope the present task was performed, is to contribute to enhanced crew and aircraft survivability in normal and hostile operational environments.

Based on data related to past aircraft fires, caused by hydraulic fluids, the Air Force established criteria for their ignitability characteristics (Ref. 1). The near term goals are designated Criteria B. The ultimate desired performance is covered by Criteria A. Encompassed in Criteria A is the requirement that the hot surface ignition temperature of fluids upon stream impingement be above 1649°C (3000°F). The need for ignition resistance at such high temperatures arises from the knowledge that the surfaces of high performance aircraft brakes attain the cited temperature upon aborted take-offs. These brake surfaces are composed of graphite composite materials. In case of hydraulic line rupture in the brake assemblies, the fluid could impinge onto the extremely hot surfaces.

An apparatus to test the ignitability of fluids on hot surfaces at 1649°C (3000°F) did not exist at the time when the Air Force performance criteria are established. The objective of the task described in this report was to design, fabricate, and test such an apparatus. The results of ignition and flame proprogation tests with a number of currently used and candidate hydraulic fluids are also described.

SECTION II

SYSTEM DESIGN AND DESCRIPTION

The following are the performance requirements and design features that were established for the apparatus:

- Surface temperatures up to 1649°C (3000°F) attainable and controllable.
- Surface of sufficiently large size, and of geometry to attain good contact with fluids.
- The angle of inclination of the surface variable from zero to ten degrees.
- Partially surrounded by an enclosure with heat-reflective walls that also control the air flow pattern.
- Access for video recording of rapidly occurring ignition events.

The total system is shown in Figure 1. It consists of the following major functional components:

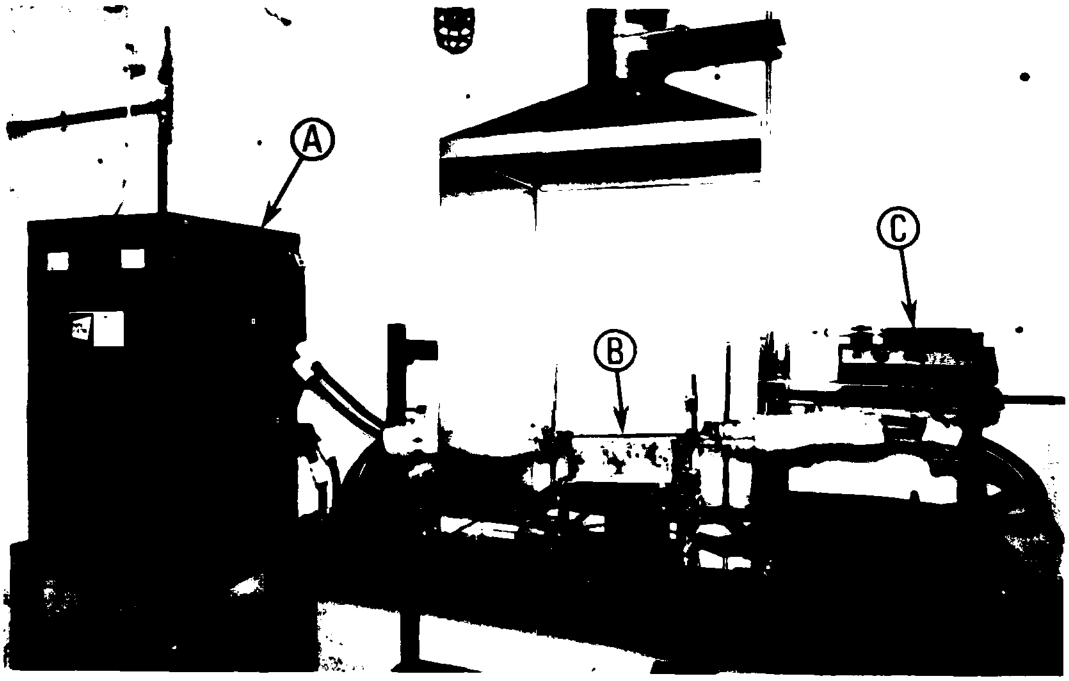
1. A consumable resistance-heated graphite heating element on a porous refractory support base, partially surrounded by an enclosure.
2. A 12 kW DC power supply.
3. Syringe pump with attached tubing for fluid delivery.

Each major component will be described in the following sections.

1. HEATER ASSEMBLY

A close-up view of the heater assembly is presented in Figure 2. A resistance-heated graphite heating element provides the high-temperature surface. These elements have been fabricated from Grade CS graphite, produced by Union Carbide Corporation, Carbon Products Division. A Tensilkut machine, normally used for the fabrication of tensil test specimens, is used for the fabrication of the graphite heating elements. A special jig was designed and fabricated for the production of the consumable heating elements.

In initial experiments, graphite heaters of the configuration shown as Design 1 in Figure 3 were used. In later experiments, a 7/8 in. length of the central section of the trough was deepened by 0.030 in. (from 0.095 in. to 0.125 in.) to increase fluid retention time and the quantity of fluid retained. A heater of modified design was used in the final stage of this test sequence.



A - power supply.

B - graphite heating element in the insulating support enclosure.

C - fluid delivery pump.

Figure 1. High temperature (1649°C, 3000°F) surface ignition test system.

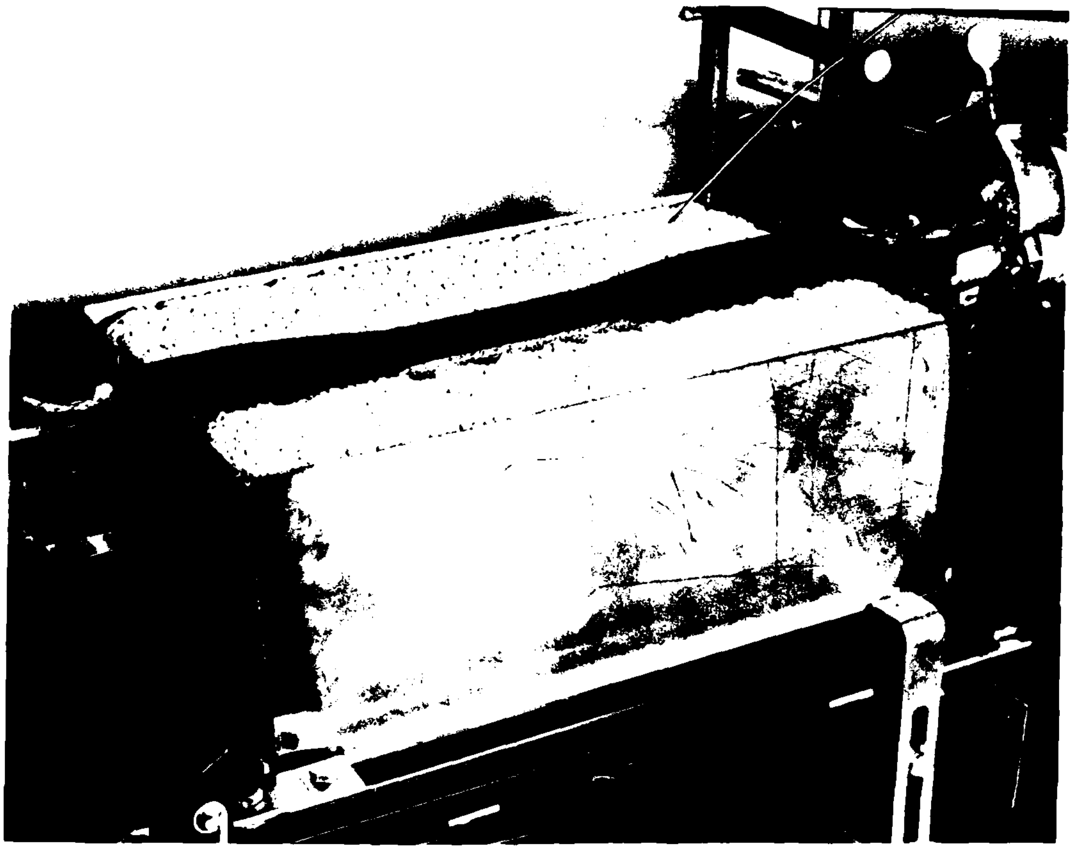
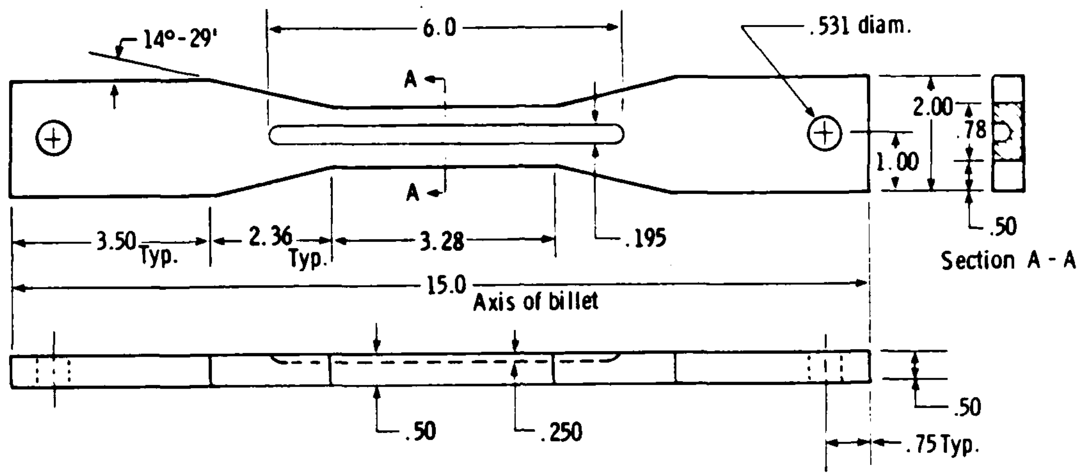
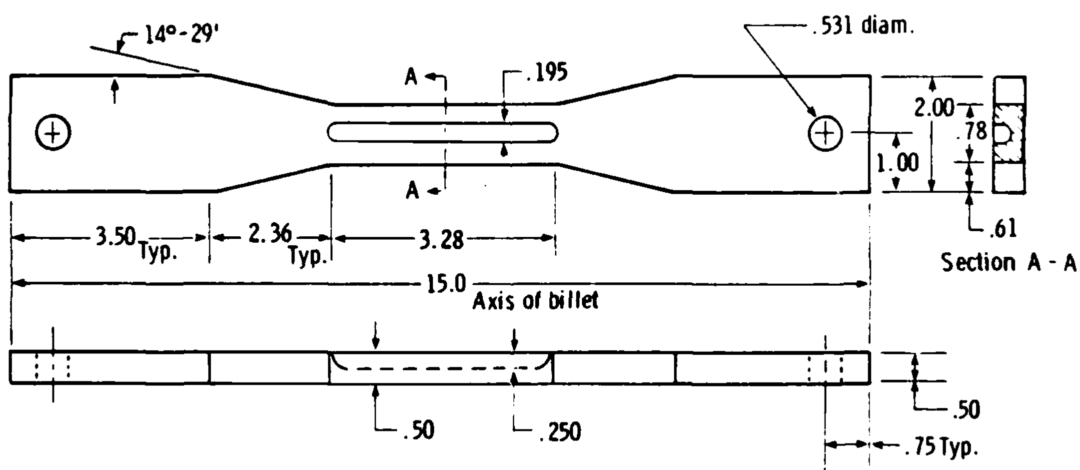


Figure 2. Graphite heater assembly.



Design 1



Design 3

Figure 3. Dimensional drawings of graphite heaters. (Dimensions in inches.)

The dimensions of the central cross section of the modified heater were changed from 0.25 in. x 1.00 in. to 0.50 in. x 0.78 in. The trough for the fluid was narrower, deeper, and shorter (0.195 in. x 0.250 in. x 3.28 in.) than that of the initially designed heater (0.50 in. x 0.095 in. x 6.00 in., respectively). The latter modification was tested to explore the feasibility of attaining more extensive heat transfer from the heater to the fluid. In the following discussion of the results, the successively used heater configurations are referred to as Designs 1, 2, and 3. In projected work, heating elements of Design 3 will be used.

The central, narrow segment of the heating element, with a longitudinal trough, provides the high-temperature surface. The fluid delivery tube is positioned above the trough, at the elevated end of the heater.

The heater rests on a low-density ceramic block, fabricated from Plicast LWI Bubble Cast (from Plibrico Company, Chicago, IL). Stainless steel fibers [Ribtec-OC (330), from RIBBON Technology Corporation, Canal Winchester, OH] were incorporated into the lower two-thirds of the ceramic casting, to increase its mechanical strength and to enhance its thermal shock resistance. The casting was made in a wooden mold, lined with Teflon. An identation was formed into the upper surface of the casting, to accommodate the graphite heater loosely.

The casting was allowed to cure in the mold at room temperature for two days. Subsequently, it was removed from the mold and heated to 100°C (220°F) in eight hours. After being maintained at that temperature overnight, the casting was heated to 204°C (400°F) in eight hours. Subsequently, for final curing, it was heated from 204°C to 816°C (400°F to 1,500°F) at the rate of 56°C (100°F) per hour.

A stainless steel enclosure was fabricated for the low-density refractory heater support, to prevent accidental breakage. The cured refractory has low thermal conductivity and high reflectance, thereby minimizing energy losses from the heater.

The stainless steel enclosure, containing the refractory heater support block, is held on a support frame fabricated from 2.06 cm (13/16 in.) Unistrut channel. The angle of inclination of the top surface of that frame can be adjusted from 0 to 10 degrees with reference to the horizontal base. It is normally maintained at 1°45'. The entire heater assembly is located in a ventilated hood that is 76.2 cm (30 in.) wide and 68.6 cm (27 in.) deep. The height of the front opening is 96.5 cm (38 in.).

2. POWER SUPPLY

A DC power supply from Rapid Electric Company, Inc., Brookfield, CT is used in the system (see Figure 4). It requires 45A, 220V, 60cps, three phase electrical power input and utilizes a silicon rectifier. This power supply is capable of providing DC output ranging up to 1,000A at 0 to 12V. Current is fed from the power supply to the graphite heater through pairs of insulated 4/0 copper welding cables. Copper lugs are used for fastening the cable to the power supply terminal plates and to the graphite heater. The four lugs in contact with the heater were goldplated by sputtering, and subsequently by electrochemical deposition (by Hohman Plating, Dayton, OH). The purpose of the gold deposit is to reduce the rate of oxidation, with associated loss of conductivity, at the heated contact surface.

To maintain 1649°C (3000°F) heating element temperature, up to 5 kW power input has been required.

3. FLUID DELIVERY

Fluid is supplied at a constant rate by means of a syringe pump (Sage Instruments, Model 355; see Figure 5). By selecting an appropriate syringe size, plunger drive rate, and internal diameter of the delivery tubing, delivery rates up to 140 mL/min can be attained. In the present experiments, the fluid was fed through a 1/16 in. (1.59 mm) OD stainless steel tubing of 0.030 in. (0.76 mm) internal diameter to a point above the surface of the graphite heater, from where it either dropped or flowed into the heated zone.

Figure 4. Power supply for the ignition test system.

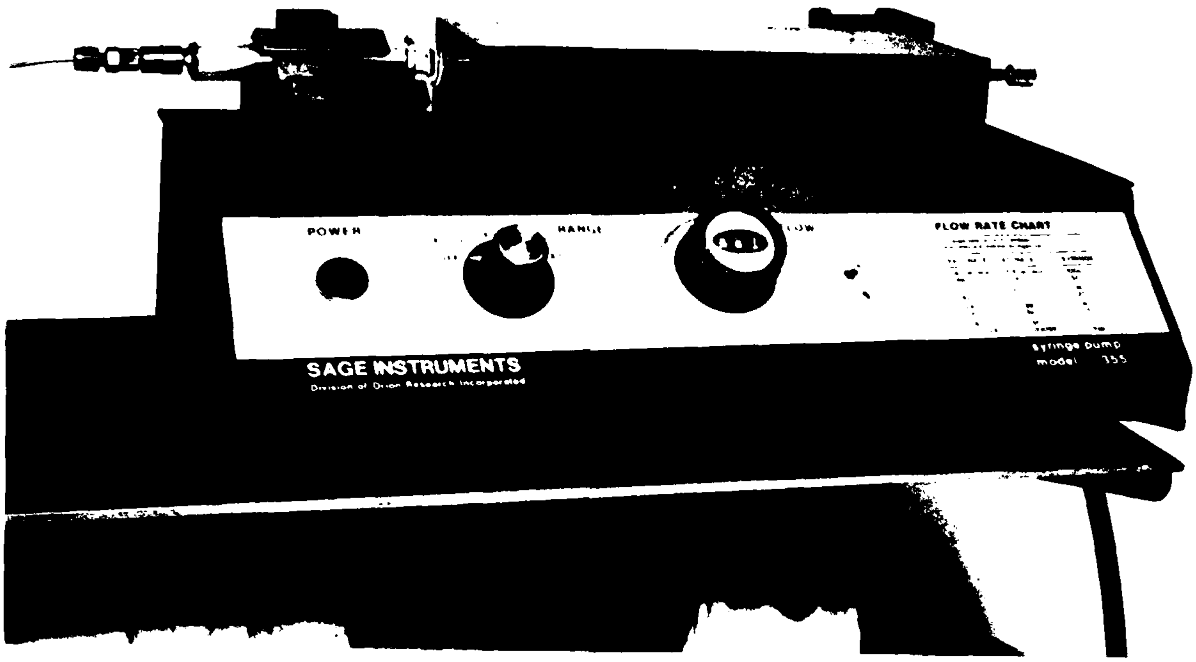


Figure 5. Fluid delivery pump.

SECTION III

EXPERIMENTAL

1. TEMPERATURE MEASUREMENT

Three temperature measurement devices were used in the experiments to be described: a chromel-alumel thermocouple, an infrared pyrometer, and an optical pyrometer. The thermocouple junction was placed into a small hemispherical cavity made into the central area of the trough. The thermocouple was connected to a Type K Digital Thermocouple Indicator from Newport Laboratories, Inc. It was used for continuous monitoring of the approximate heater surface temperature up to 927°C (1,700°F). To avoid junction burnout, the thermocouple was not used at higher temperatures. Because of loose contact between the thermocouple and the heater surface, the temperatures determined in this manner were not accurate.

The Heat Spy infrared pyrometer, Model HSA-6E, was used for temperature measurement in the range up to 1,000°C (1,832°F). An emissivity setting of 0.75 was used on the infrared pyrometer for graphite during these measurements.

The Leeds and Northrup disappearing wire type optical pyrometer, Model 8622-C, was used for surface temperature measurements at temperatures above 1,000°C (1,832°F). The infrared and the optical pyrometer were both recalibrated by their respective manufacturers before the test sequence described here was initiated.

2. OPERATIONAL TESTS AND PERFORMANCE

The required surface temperature is reached by progressively increasing electrical power input into the graphite heater. The surface temperature of 1649°C (3000°F) is attained in 10 to 15 minutes after start-up. Figure 6 depicts the system in operation, with the heater surface at 1649°C (3000°F). The presently required maximum surface temperature was attained at approximately one half of the rated output of the power supply.

The length of the uniformly heated zone on the graphite bar is approximately 7.6 cm (3 in.). It is of interest to note that the desired operating temperature, once attained, can be maintained readily by minor adjustments of powerstat settings.

Graphite is known to be oxidized in air at high temperatures (Ref. 2). Graphite-containing composites (e.g., in aircraft brake surfaces) can be expected to react analogously.

A pale blue flame was observed above the surface of the graphite heating element, arising from the oxidation of graphite, at temperatures above 677°C/1,250°F (see Figures 6 and 7). In oxidation resistance, the graphite heating element simulates the graphite composite surfaces of aircraft brake assemblies.

Figure 6. Graphite heating element, with surface at 1649°C (3000°F).

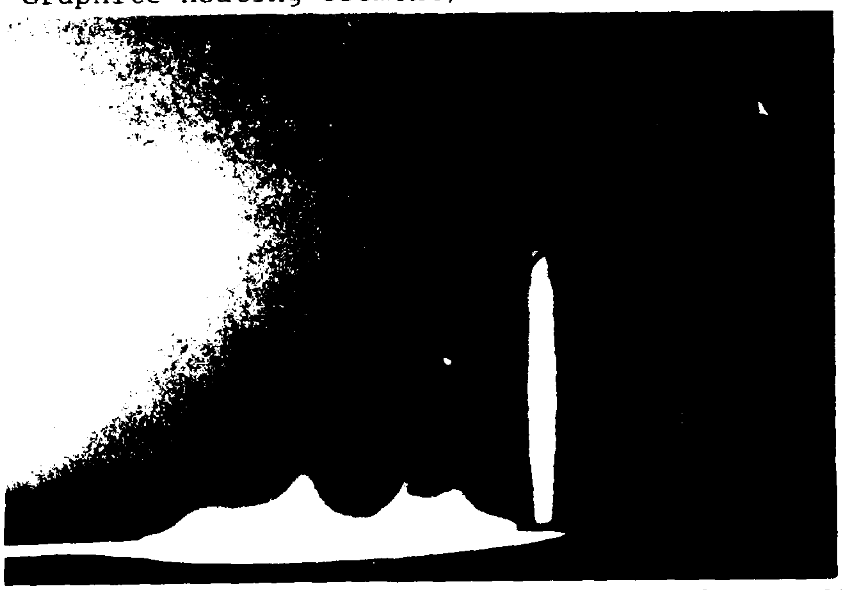


Figure 7. Flame above the heater surface at 1649°C (3000°F). [To enhance the visibility of the pale blue flame above the heated graphite surface, this photograph was taken through a dark blue (81A) filter.]

While the oxidative erosion of the graphite causes progressive decrease of current flow, and relatively smaller changes of potential, the heater surface temperature changes little. The experimental record from a heating cycle, in which the service life of the graphite heater was determined, is presented in Table 1.

Using data collected during experiments, working graphs were prepared that depict heater surface temperature as a function of applied potential (see Figure 8). Successively higher potential had to be applied to attain a desired temperature as the heaters were used, especially at high temperatures.

3. FLUIDS

Samples of MIL-H-5606 and MIL-H-83282 hydraulic fluids were received in sealed 1-gallon containers from the Fire Protection Branch, AFWAL, for the present test sequence.

One quart samples of Halocarbon AO-2 (MLO 81-509) and Freon E6.5 (MLO 77-85) were received from Dr. C. E. Snyder (AFWAL/MLBT). Halocarbon Products Corporation produces a class of chlorotrifluoroethylene (CTFE) fluids which are the prime candidates for the Air Force nonflammable hydraulic development program. These fluids are designated Halocarbon AO-2 or AO-8. The only difference is the molecular weight and, hence, the viscosity. The flammability characteristics of either AO-2 or AO-8 are expected to be the same. Samples were designated as MLO 81-509 (AO-2) and MLO 76-74 (AO-8). Designation for this report will be CTFE, consistent with Air Force terminology.

Also, samples of a perfluoroalkyl ether fluid, produced by Bray Oil Company and identified as Bryco 814Z were available from previous work. They were designated MLO 76-107 and MLO 78-80.

Nadraul MS-6 had been supplied to the Air Force by Mr. A. Conte of the Naval Air Development Command. This sample carried the identification MLO 77-41.

Skydrol 500B, produced by Monsanto Company, was part of a sample that had been purchased for tests performed previously (Ref. 1).

4. TESTING PROCEDURE

The resistance-heated graphite bar was brought to the desired, predetermined temperature by controlling the applied electrical potential with the power supply. The heating element and its insulating support were allowed to equilibrate thermally.

Fluid delivery was initiated after the test system had reached thermal equilibrium. Two delivery rates (0.6 mL/min and 6 mL/min) were used in present experiments. The delivery time was

TABLE 1. OPERATIONAL DATA FOR THE HIGH-TEMPERATURE IGNITION TEST APPARATUS[a]

Time, min	Powerstat setting	Potential, V	Current, A	Power, kW	Surface temperature, °C	Surface temperature, °F	Observations
0							Heating started.
3	19	2	240	0.48			
4	26	3	370	1.10	880	1,616	Heater appeared red.
7	32.5	4	470	1.88	1,120	2,048	
9	39.6	5	550	2.20	1,323	2,413	Blueish flame appeared over the graphite surface.
11	45	6	580	3.48	1,432	2,610	Oxidative surface-erosion was visible.
12		7	640	4.48	1,542	2,808	
13	57	8	680	5.44	1,645	2,993	Uniformly heated zone was ~3 in. long. Blueish flames appeared over this entire length.
16	57				1,675	3,047	Power input reduced.
17	55.5	8	625	5.00	1,652	3,006	
18	55.5	8	595	4.76	1,647	2,997	
20	55.5	8			1,662	3,024	Power input reduced.
21	54.5	8			1,637	2,979	
22	54.5	8			1,658	3,018	
24	54.5	8.05	480	3.86	1,649	3,000	
26	54.5	8.0	455	3.64	1,649	3,000	
28	53.5	8.0	420	3.36	1,649	3,000	
32	53.5	8.2	335	2.75	1,649	3,000	
35	53.5	8.35	280	2.34	1,649	3,000	
38	53.5	8.35	210	1.80	1,649	3,000	
40	53.5	8.75	150	1.31	1,649	3,000	One side of the heater central segment burned through.

[a]These data were acquired with a graphite heating element of Design 1.

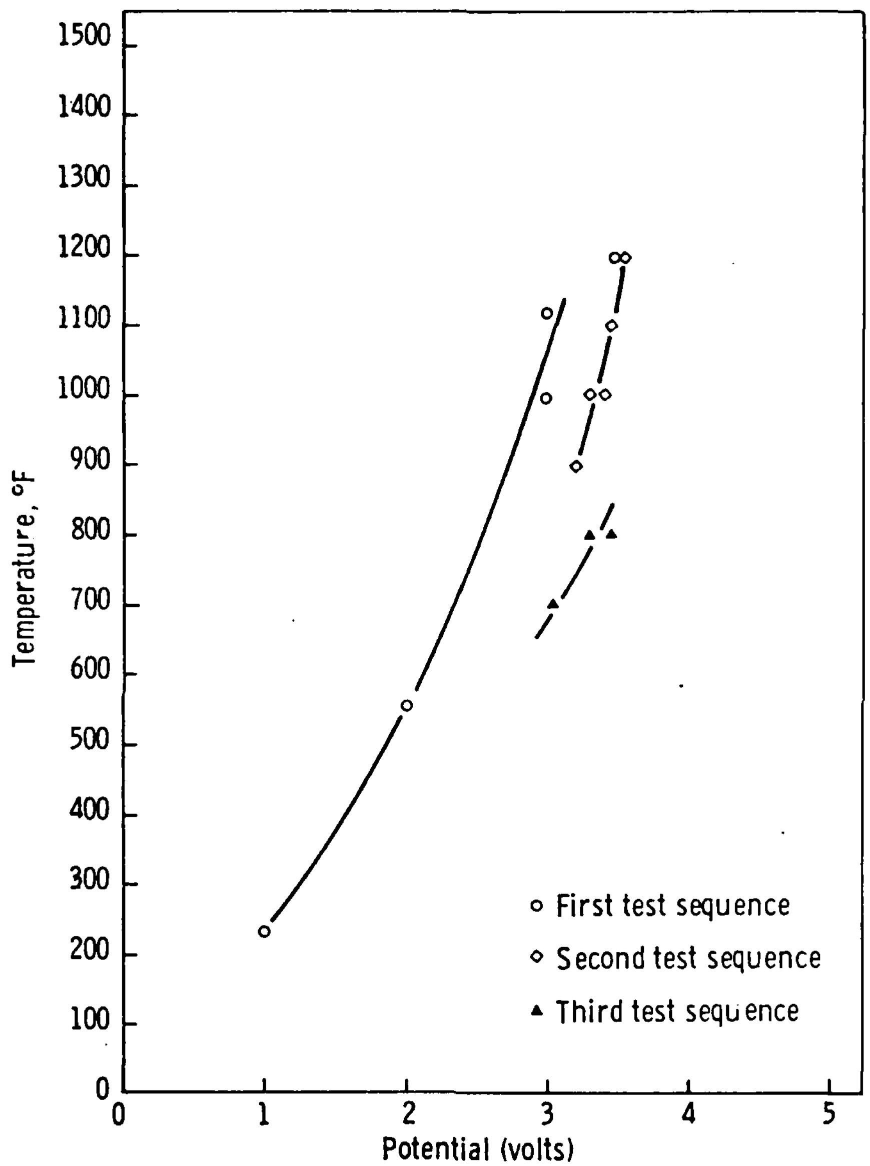


Figure 8. Surface temperature of a graphite heater as a function of applied potential and prior use.

one minute, unless ignition occurred sooner. In the latter case, fluid delivery was terminated after the needed observations had been made.

If thermal degradation or combustion of a fluid produced a residue, it was removed prior to proceeding to tests under altered conditions.

SECTION IV

RESULTS AND DISCUSSION

The following two types of ignition tests were performed:

1. Qualitative observations of ignitability and flame propagation behavior of fluids upon impingement onto graphite surface heated to 1649°C (3000°F).

2. Determination of minimum ignition temperatures.

The results of both types of tests will be described.

1. IGNITABILITY OF FLUIDS AT 1649°C (3000°F) AND FLAME PROPAGATION

The hydrocarbon type fluids ignited very readily, as would be expected on the basis of previous work (Ref. 1 and publications cited in that report). The mineral oil-based hydraulic fluid (MIL-H-5606) vaporized more rapidly than the synthetic fluid (MIL-H-83282). Therefore, more extensive flame propagation was noted with the former fluid. At 6 mL/min delivery rate, the flames progated up to the tip of the delivery tube and continued to engulf it with MIL-H-5606. Only occasional propagation to that point was observed with the synthetic hydrocarbon fluid (see Figure 9). The splattering droplets of the mineral oil-based fluid propagated flames much more extensively than those of the other hydrocarbon fluid under the conditions of this test.

The fluorinated candidate hydraulic fluids were very resistant to combustion upon impingement onto the 1649°C (3000°F) surface. Close-up photographs in Figure 9 depict the extensive aerosolization and splattering of these fluids from the hot surface, when delivered at the rate of 0.6 mL/min. At faster delivery rates, almost all of the delivered fluid was evaporated and subsequently condensed to a cloud of aerosol in the exhausted hood.

The flame structure associated with the burning of hydrocarbon fuels is well-defined in Figure 9. Only low flames were observed with CTFE-based fluids and with the fluoroalkyl ether. The diffuse-appearing light areas in Figure 9, observed in tests with the latter fluids, were caused by the aerosol clouds. No ignition was observed with the perfluoroalkyl ether. No significant difference was detected in performance between the formulated and unformulated CTFE fluids.

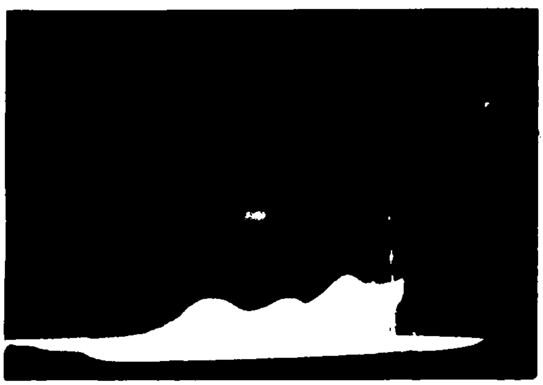
The introduction of the fire-resistant fluids into the high temperature environment above the graphite surface caused a change of the flame color from pale blue to yellow. The flames arising from the fluids propagated very short distances, extending only up to three inches downstream from the point of impingement, and approximately

MIL-H-5606

MIL-H-83282

Formulated CTFE

Unformulated CTFE



Fluoroalkyl ether (Freon E6.5)

Perfluoroalkyl ether (Brayco 814Z)

Figure 9. Ignition tests with presently used and candidate hydraulic fluids.

the same distance upward. Under some test conditions (rapid flow with CTFE fluids), soot formation became observable.

Fire resistance of the two highly fluorinated ether type fluids is demonstrated dramatically in Figure 10. Droplets of the fluids can be seen splattering from the 1649°C (3000°F) surface. Low flames were visible above the graphite surface when the fluoroalkyl ether fluid was allowed to impinge upon it; the perfluoroalkyl ether functioned as an extinguishing agent for the low blue flames emanating from the graphite surface.

On the basis of these observations, the relative propensity of fluorinated candidate hydraulic fluids for ignition under the conditions of this test is ranked as follows:

Perfluoroalkyl ether < fluoroalkyl ether < CTFE
(Brayco 814Z) (Freon E6.5)

As described above, visual evidence was obtained for high-temperature reactions with the three fluorine-containing organic fluid compositions: fluoroalkyl ether, and formulated and unformulated CTFE. These reactions would entail pyrolysis and oxidation. The products from both types of reactions may be toxic. Therefore, caution is advised in work with these thermally stable fluids under extreme high temperature conditions.

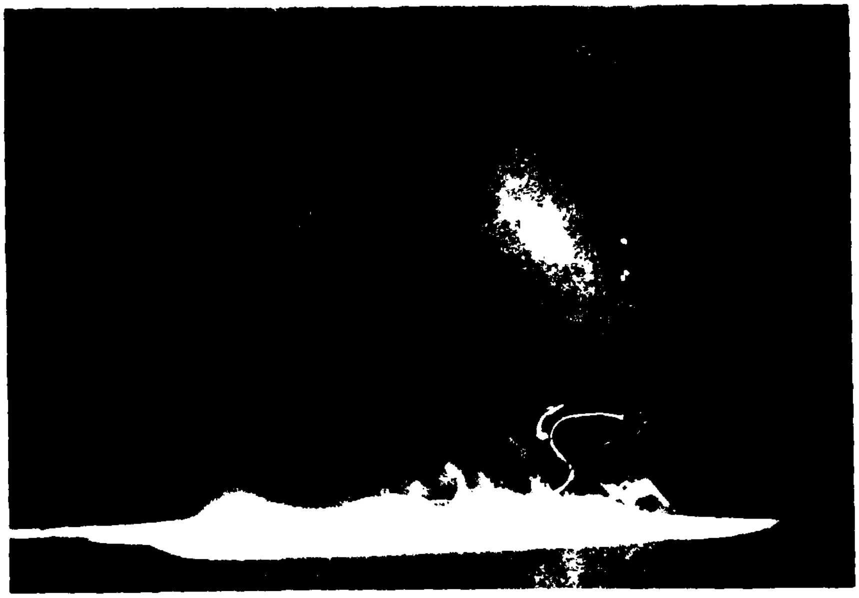
2. IGNITABILITY EXPERIMENTS WITH FLUIDS AT TEMPERATURES RANGING UP TO 1649°C (3000°F)

The experimental results are included as the Appendix to this report. The data pertaining to ignitability of the fluids are summarized in Figure 11. The surface temperatures of the graphite heater, at which ignition of impinging fluids was observed, are indicated in this figure by filled circles. A comparison of the minimum ignition temperatures of fluids on graphite heater with those on the hot manifold, and with the autoignition temperatures determined by the ASTM procedure, is provided in Table 2.

The data indicate that the rate of fluid delivery had no significant effect upon the ignition temperature, within the range of rates included in the present experiments.

a. Hydrocarbon Type Fuels

The ignition temperatures of hydrocarbon fluids (MIL-H-5606 and MIL-H-83282) on the graphite surface were approximately 194°C (350°F) higher than the values determined with the hot steel manifold (see Table 1). Two changes were made in trough configuration, to increase fluid retention time and exposure of vapors to the heater walls. These had a small effect on the ignition temperature. Subsequently, the temperature of liquid droplets (at 0.6 mL/min fluid delivery rate) and puddles (at



Fluoroalkyl ether ([illegible] F6.[illegible])

Perfluoroalkyl ether (Brayco 814Z)

Figure 10. Fluid droplets splattering from the hot surface (1649°C, 3000°F) during slow delivery (0.6 mL/min).

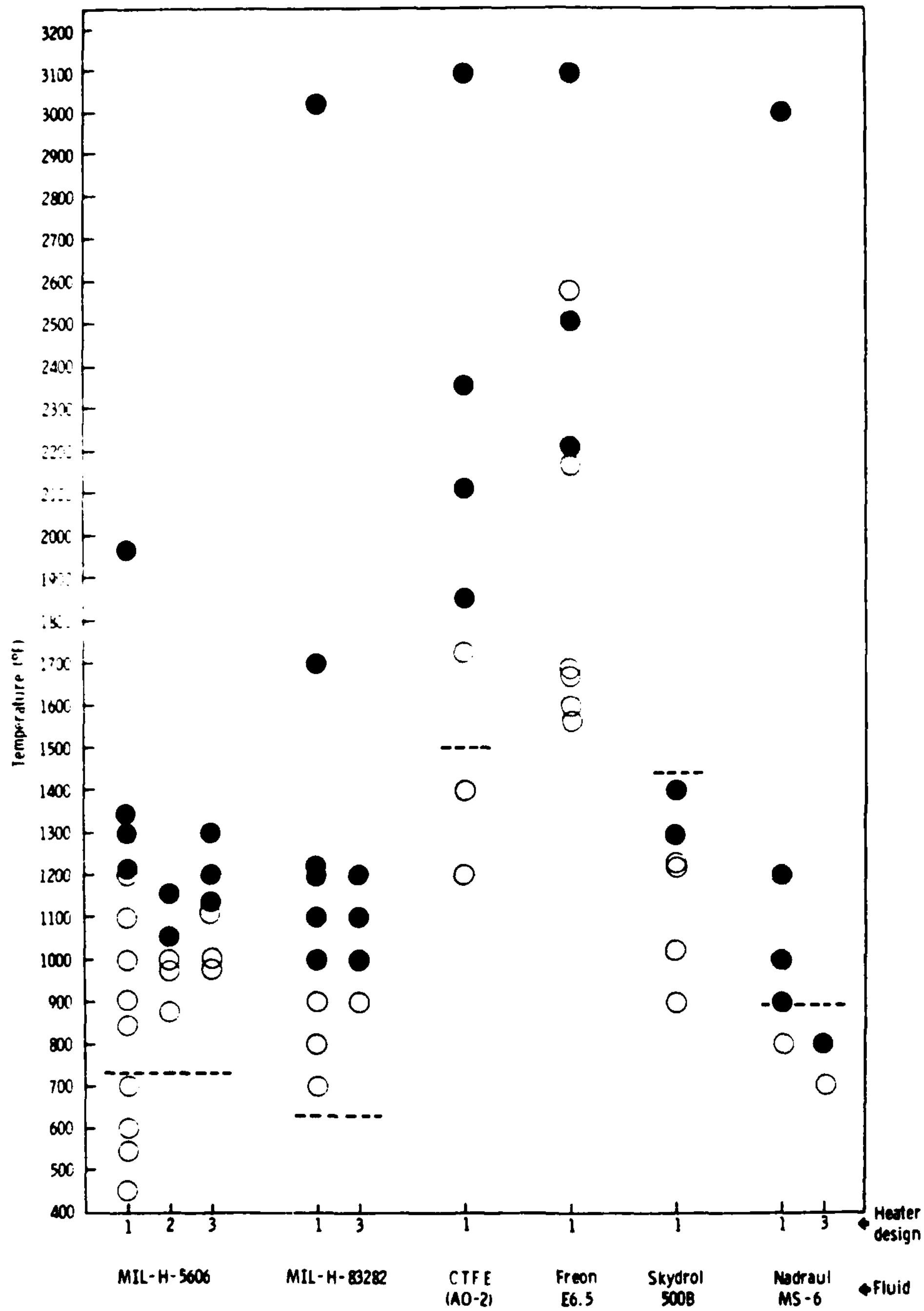


Figure 11. Ignition test results for fluids on graphite heater. (Filled circles indicate temperatures at which fluids ignited on graphite surface; unfilled circles indicate test temperatures at which ignition did not occur. Dashed lines represent ignition temperatures for liquid stream delivery on hot steel manifold).

TABLE 2. SUMMARY OF IGNITION TEMPERATURES

Fluid	Ignition temperatures, °C			Ignition temperatures, °F		
	AIT[a]	Hot manifold, stream delivery[a]	Graphite heater	AIT[a]	Hot manifold, stream delivery[a]	Graphite heater
MIL-H-5606	238	388	570[b]	461	730	1,058
MIL-H-83282	347	322	538[c]	656	630	1,000
CTFE	630	816	1,010	1,165	1,500	1,850
Freon E6.5	669	>927	1,210	1,235	>1,700	2,210
Skydrol 500B	510	782	704	950	1,440	1,300
Nadraul MS-6	409	477	427	770	890	800

[a]Data from Reference 1.

6.0 mL/min delivery rate), that were in contact with the thermocouple, was monitored during the experiments. These measurements were made with the hydrocarbon fluids impinging onto the surface of the heater that contained the narrow, deep trough (Design 3). The results of these measurements are shown in Figures 12 and 13.

The fluid temperatures were found to be significantly [up to ~371°C (700°F)] lower than the respective temperatures of the heater surface, especially for the more volatile fluid (MIL-H-5606). That fluid was cooled more extensively by vaporization. It was also learned that the temperatures of the fluids on the graphite heater surface, below the surface temperature at which ignition occurred, were significantly higher than the minimum autoignition temperatures determined in a Vycor flask by the ASTM procedure.

The above-cited data and observations led to two conclusions regarding fluids whose vapor pressures are sufficiently high to allow significant vaporization below the ignition temperature:

(1) The temperature of the liquid in contact with the heating surface in dynamic tests (such as hot manifold and graphite surface ignition tests) can be much lower than the temperature of the surface.

(2) The temperatures, temperature gradients, volumes of the heated vapor zones, and the flow patterns of vapors at the heated surface have a pronounced effect on the observed ignition temperature.

The differences in ignition temperatures for the hydrocarbon type fluids, as determined with the hot manifold and the graphite surface ignition test systems, are attributed to the factors cited in the latter conclusion. In tests conducted with the graphite heater system, fuel vapors leave the heated zone. With the manifold test system, the heated zone volume is larger and vapors have more extensive contact with the heated surface. Consequently, ignition temperatures determined with the latter system are lower than those measured with the graphite heater (high temperature) system.

b. Nonhydrocarbon Fluids

Two halogen-containing fluids in the present work (CTFE (AO-2) and Freon E6.5) are thermally very stable over an extensive temperature range. Their ignition temperatures under all test conditions would be relatively high; their heats of combustion are very low (<3,100 BTU/lb; Ref. 1). They burn much less vigorously than the fluids that have heats of combustion in the 9,000 BTU/lb to 21,000 BTU/lb range.

The oxidation of the graphite heater becomes detectable at temperatures above 677°C (1,250°F). During the present work, the lowest

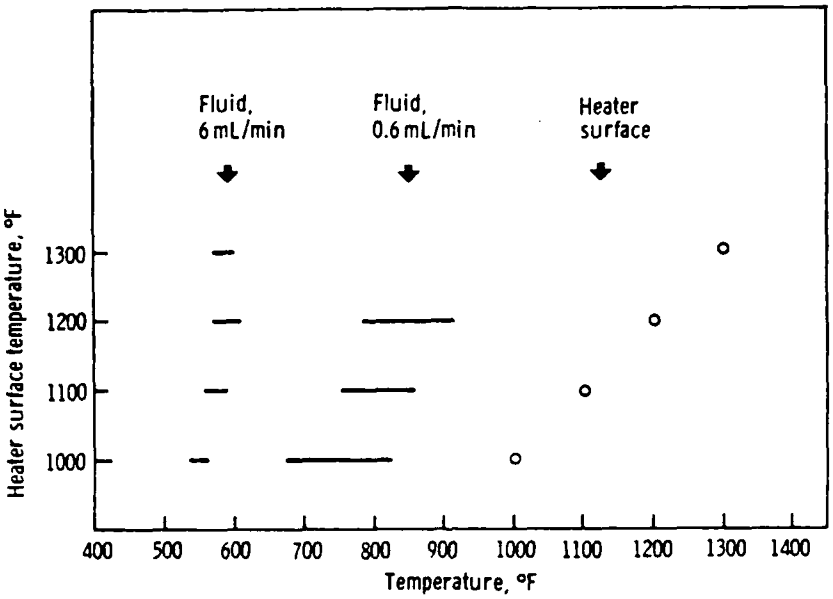


Figure 12. Temperature of MIL-H-5606, in contract with the thermocouple on heater surface.

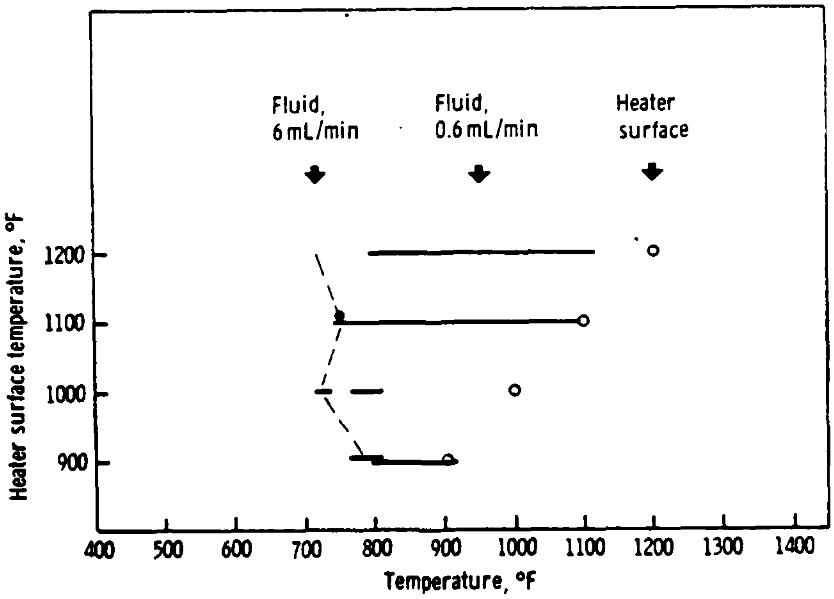


Figure 13. Temperature of MIL-H-83282, in contact with the thermocouple on heater surface.

temperature at which very weak bluish flamelets (≦5 mm high) were observed in an unlighted laboratory was 700°C (1,292°F). The bluish flame is present on the graphite surface at temperatures at which the two halogen-containing fluids would ignite spontaneously on inert surfaces. Because of the presence of that flame, and the low intensity that characterizes the burning of these fluids, it is not feasible to define precisely the temperature at which they ignite on the graphite surface. Furthermore, both fluids were found to function as retardants for the slow burning of graphite, at temperatures ranging up to 1,427°C (2,600°F), when delivered at the rate of 6 mL/min (see Table A-3, Test Sequence 7, and Table A-4, Test Sequences 4 and 5).

At high surface temperatures, thermal activation and degradation of the fluids molecules occurs to a sufficient extent to give rise to combustion. The temperatures in Figure 11, at which ignition of CTFE(AO-2) and Freon E6.5 is indicated, are those at which a definite contribution to the flames caused by the burning of graphite was observed.

Although CTFE and Freon E6.5 fluids do burn when they impinge onto a very hot surface, it is important to note that the flame intensities are very much lower than those associated with the burning of all other fluids used in the present test sequence.

Nadraul MS-6 and Skydrol 500B ignited on the graphite heater at ~55°C (100°F) lower temperature than on the heated steel manifold. With the first of these fluids, extensive wetting of the surface occurred. At low temperatures, the burning was intermittent. Small bursts of flames, emanating from the heater surface, were observed. It appeared that the ignition was associated with thermal or thermo-oxidative degradation of the fluid.

The ignition of Skydrol 500B on the graphite heater, at temperatures below those at which ignition occurred on the steel manifold, is attributed to flames present on the graphite heater at temperatures above ~677°C (1,250°F). Ignition of this fluid was not observed below temperatures at which onset of graphite oxidation had been observed.

SECTION V

CONCLUSIONS

1. The high temperature (graphite surface) ignition test apparatus has been found to meet the performance requirements established prior to its design. This apparatus is a useful, laboratory-scale system for the evaluation of ignition and flame propagation characteristics of fluids over a very wide temperature range (up to 1,649°C [3,000°F]). In particular, the apparatus simulates ignition conditions on graphite and graphite composite surfaces (e.g., brake assemblies of high performance aircraft).

2. At temperatures above 677°C (1,250°F), ignition of flammable fluids can be caused by the low flames on the graphite surface, arising from its oxidation at high temperatures. The same ignition mechanism may be operative when readily ignitable fluids impinge onto graphite composite brake surfaces.

3. Ignition temperatures of fluids on graphite heater surface were found to range from 78°C (140°F) below to 206°C (370°F) above those determined previously with the steel manifold system. Ignition temperatures higher than those observed with the steel manifold are attributed to the following factors:

   (1) Volatility characteristics of the fluids

   (2) Temperatures, temperature gradients, and volumes of the heated vapor zones

   (3) Flow patterns of vapors at the heated surfaces.

4. Ignition temperatures of fluids on the hot steel manifold test system can be estimated from data obtained with graphite heater ignition test apparatus on the basis of presently reported results, and our understanding of factors that control ignitability. We estimate that the predicted values for hot manifold ignition temperatures will be within ±111°C (200°F) of the experimental values.

5. Ignition data obtained with the graphite heater test apparatus, combined with autoignition test data (by ASTM Method D2155), and heat of combustion data provide a useful means for laboratory-scale evaluation of the ignitability and flammability of fluids in potential hazard situations. The AIT data indicate lowest temperatures at which ignition of fluids could be anticipated in a static system (quiescent atmosphere, where the vaporizing fluid and air mix thoroughly in a small volume by slow diffusion). The ignition temperatures determined with the hot surface test apparatus pertain to dynamic systems of small size, in which the temperature gradients in the vapor phase are high, and the heated vapors are rapidly removed from

the area of the heat source. The heat of combustion data allow concerned persons to develop a qualitative appreciation of the potential severity of unwanted fires.

REFERENCES

1. L. Parts, "Assessment of the Flammability of Aircraft Hydraulic Fluids," AFAPL-TR-79-2055, July 1979.

2. J. M. Thomas, "Reactivity of Carbon. Some Current Problems and Trends," Carbon 8, 413(1970).

APPENDIX

EXPERIMENTAL DATA

TABLE A-1. IGNITION TEST RESULTS WITH MIL-H-5606

Test sequence number	Manifold temperature °C	Manifold temperature °F	Heater design[a]	Fluid delivery rate, mL/min	Ignitability characteristics[b]	Observations
1	190-232	374-450	1	0.6	NI, NI, NI, NI, NI	
2	240-285	464-545	1	6.0	NI	
3	316	600	1	0.6	NI, NI	
4	371	700	1	6.0	NI	Fluid droplets beaded on graphite surface.
5	450	842	1	0.6	NI, NI	
6	482	900	1	0.6	NI	
7	482	900	1	6.0	NI, NI	
8	538	1,000	1	0.6	NI	Graphite heater dull red in the center.
9	538	1,000	1	6.0	NI	Beading, puddling and extensive vaporization of the liquid.
10	593	1,100	1	0.6	NI	
11	593	1,100	1	6.0	NI	
12	649	1,200	1	6.0	NI	
13	655	1,211	1	0.6	I	Droplets ignited upon impingement onto graphite surface.
14	655	1,211	1	6.0	I	Continuous burning after ignition. Flames extended up to ~14 in. above the heater surface.
15	704	1,300	1	0.6	I, I, I	Flames extended 3 in. to 4 in. above the heater surface.
16	704	1,300	1	6.0	I, I, I	Flames extended up to ~12 in. above the heater surface.
17	730	1,346	1	0.6	I	
18	730	1,346	1	6.0	I	Flames extended up to ~14 in. above the heater surface.
19	1,075	1,967	1	6.0	I	Flames only ~15% higher than at 1,300°F.

(continued)

TABLE A-1 (continued)

Test sequence number	Manifold temperature °C	Manifold temperature °F	Heater design[a]	Fluid delivery rate, mL/min	Ignitability characteristics[b]	Observations
20	470	878	2	6.0	NI	
21	525	977	2	0.6	NI	
22	538	1,000	2	0.6	NI, NI, NI, NI	
23	538	1,000	2	6.0	NI	
24	570	1,058	2	0.6	I	
25	625	1,157	2	0.6	I, I, I	
26	630	1,166	2	6.0	I	Ignition occurred after fluid delivery had been completed.
27	527	981	3	0.6	NI, NI, NI	
28	538	1,000	3	6.0	NI, NI, NI	
30	593	1,100	3	0.6	NI, NI, NI	
31	593	1,100	3	6.0	NI, NI, NI	
32	600	1,112	3	0.6	NI, NI, NI	
33	615	1,139	3	6.0	NI, I, NI	Ignition occurred after fluid delivery had been completed.
34	649	1,200	3	0.6	I, NI, NI	Ignition delay time was 8 seconds.
35	649	1,200	3	6.0	NI, I, NI	Ignition delay time was 4 seconds.
36	704	1,300	3	0.6	I, I, I	Ignition occurred during initial impingement of fluid.
37	704	1,300	3	6.0	NI, I, I	Ignition occurred during initial impingement of fluid.

[a]See apparatus description in the Experimental section.

[b]NI - no ignition.
I - ignition occurred during this test.

TABLE A-2. IGNITION TEST RESULTS WITH MIL-H-83282

Test sequence number	Manifold temperature °C	Manifold temperature °F	Heater design	Fluid delivery rate, mL/min	Ignitability characteristics	Observations
1	371	700	1	0.6	NI	
2	371	700	1	6.0	NI	Fluid evaporated completely.
3	427	800	1	0.6	NI	
4	427	800	1	6.0	NI	
5	482	900	1	0.6	NI	
6	482	900	1	6.0	NI	Fluid beaded upon impingement onto heater surface, evaporated completely.
7	538	1,000	1	0.6	I, NI	
8	538	1,000	1	6.0	I, NI	
9	593	1,100	1	0.6	I, I	Ignition delay times 33 seconds and 1 second, respectively.
10	593	1,100	1	6.0	I	Ignition delay time 3 seconds.
11	649	1,200	1	0.6	I	Each droplet ignited, as it impinged onto the heater surface.
12	649	1,200	1	6.0	I	
13	660	1,220	1	0.6	I	
14	660	1,220	1	6.0	I	Flames extended up to 7 in. above the heater surface.
15	927	1,700	1	0.6	I	
16	927	1,700	1	6.0	I	Flames were not much higher than in the 1,200°F experiments.
17	1,660	3,020	1	0.6	I	
18	1,660	3,020	1	6.0	I	Flames extended 9 in. to 10 in. above heater surface.
19	482	900	2	0.6	NI, NI, NI	
20	492	900	2	6.0	NI, NI, NI	
21	538	1,000	2	0.6	NI, NI, NI	
22	538	1,000	2	6.0	I, I, NI	Ignition delay times 20 seconds and 4 seconds, respectively.
23	593	1,100	2	0.6	I, NI, I	Ignition delay times 4 seconds and 60 seconds, respectively.
24	593	1,100	2	6.0	I, I, I	Ignition delay times 5 seconds, 6 seconds, and 4 seconds, respectively.
25	649	1,200	2	0.6	I, I, NI	Ignition delay times 5 seconds and 60 seconds, respectively.
26	649	1,200	2	6.0	I, I, I	Ignition delay times 8 seconds, <1 second, and <1 second, respectively.

TABLE A-3. IGNITION TEST RESULTS WITH CTFE(AO-2)

Test sequence number	Manifold temperature °C	Manifold temperature °F	Heater design	Fluid delivery rate, mL/min	Ignitability characteristics	Observations
1	649	1,200	1	0.6	NI	Droplets bounced in the heater trough.
2	760	1,400	1	0.6	NI	
3	940	1,724	1	0.6	NI	Fluid vaporized.
4	1,010	1,850	1	0.6	I	Infrequent flashing, extending over a part of the surface, was observed. The surface-flame, caused by the oxidation of graphite, appeared lighter blue-colored in the presence of Halocarbon AO-2.
5	1,155	2,111	1	~1.0	I	Intermittent, weak flashing over a part of the heater surface.
6	1,290	2,354	1	0.6	I	More frequent flashing, low flamelets.
7	1,290	2,354	1	6.0	NI	Flashing not observable.
8	1,700	3,092	1	0.6	I	Continuous flaming; yellow flame was only ~0.4 in. high.
9	1,700	3,092	1	6.0	I	Continuous flaming. The flame was not much higher than at 0.6 mL/min delivery rate.

TABLE A-4. IGNITION TEST RESULTS WITH FREON E6.5

Test sequence number	Manifold temperature °C	Manifold temperature °F	Heater design	Fluid delivery rate, mL/min	Ignitability characteristics	Observations
1	850	1,562	1	6.0	NI	The low, bluish flames from graphite oxidation completely extinguished by Freon E6.5.
2	871	1,600	1	0.6	NI	The low, bluish flames from graphite oxidation reduced in intensity by Freon E6.5 at slow delivery rate.
3	910	1,670	1	0.6	NI	Fluid vaporized extensively. The low, bluish flames from graphite oxidation reduced in intensity.
4	920	1,688	1	6.0	NI	The low, bluish flames from graphite oxidation completely extinguished by Freon E6.5.
5	1,185	2,165	1	6.0	NI	Flames from graphite oxidation completely extinguished by Freon E6.5. Graphite surface cooled.
6	1,210	2,210	1	0.6	I	The intensity of the low flame from graphite oxidation increased slightly by Freon E6.5 at slow delivery rate.
7	1,375	2,507	1	0.6	I	Very extensive vaporization of the fluid. Flame height associated with graphite oxidation ~0.6 in. Increased to ~1 in. upon impingement of Freon E6.5. Color of flame changed from bluish to yellowish-blue.
8	1,415	2,579	1	6.0	NI	Fluid puddle formed in the trough. Fluid evaporated very extensively. The bluish flames arising from graphite oxidation were not completely extinguished by Freon E6.5 vapors.
9	1,700	3,092	1	0.6	I	Yellowish-blue flames.
10	1,700	3,092	1	6.0	I	The flames from graphite oxidation were affected by the impinging and evaporating fluid. The flames appeared yellowish close to the graphite surface. Wisps of weak flamelets extended up to ~4 in. above graphite surface.

TABLE A-5. IGNITION TEST RESULTS WITH SKYDROL 500B

Test sequence number	Manifold temperature °C	Manifold temperature °F	Heater design	Fluid delivery rate, mL/min	Ignitability characteristics	Observations
1	482	900	1	0.6	NI	Extensive evaporation and thermal degradation; black char formed.
2	550	1,022	1	0.6	NI	
3	660	1,220	1	0.6	NI	
4	660	1,220	1	6.0	NI	
5	667	1,232	1	0.6	NI	Very extensive vaporization and degradation.
6	667	1,232	1	6.0	NI	Very extensive vaporization and degradation.
7	704	1,300	1	0.6	I	Blue flamelets intermittently visible on graphite surface before fluid delivery. Ignition delay time for fluid ignition <5 seconds.
8	760	1,400	1	0.6	I, I, I	Bluish flames continuously visible on graphite surface before fluid delivery. Skydrol 500B ignited immediately upon impingement onto heater surface. Flame height was ≤6 in.
9	760	1,400	1	6.0	I	Flames extended up to 13 in. above the graphite heater surface.

TABLE A-6. IGNITION TEST RESULTS WITH NADRAUL MS-6

Test sequence number	Manifold temperature		Heater design	Fluid delivery rate, mL/min	Ignitability characteristics	Observations
	°C	°F				
1	427	800	1	0.6	NI	Extensive vaporization and thermal degradation of the fluid. Non-volatile degradation products formed a gum in the heater trough.
2	482	900	1	0.6	I	
3	482	900	1	6.0	I	
4	538	1,000	1	0.6	I	Maximum flame height ~1.5 in.
5	538	1,000	1	6.0	I	Slowly burning liquid puddle formed in the trough. Maximum flame height ~4 in.
6	649	1,200	1	6.0	I	Maximum flame height ~6 in. Burning fluid formed a glowing char.
7	1,649	3,000	1	6.0	I	Fluid burned intensely. Flames extended up to ~12 in. above the heater surface.
8	371	700	3	0.6	NI, NI	Slow vaporization of the fluid.
9	427	800	3	0.6	I, I, I	
10	427	800	3	6.0	I, I, I	Intermittent flashing, apparently associated with the formation of thermal degradation products and their accumulation in the vapor phase.

*USGPO: 1983-659-062-1094

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